

Research on the mechanical and thermal properties of potting adhesive with different fillers of h-BN and MPCM

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ARTICLE INFO

Handling Editor: Huihe Qiu

Keywords:

Potting adhesive
Thermal behavior
Mechanical
h-BN
MPCM

ABSTRACT

Using packaging materials to reduce contact thermal resistance has become a promising method to solve the problem of insufficient heat dissipation capacity of electronic components. The purpose of this work is to optimize the mechanical and thermodynamic performance of potting adhesive using phase change microcapsules (MPCM) and hexagonal boron nitride nano-powder (h-BN) as thermal conductive fillers. The experimental results indicated that h-BN has a positive effect on the tensile strength of the potting adhesive, with a 7.1 % increase in tensile strength at a mass fraction of 30 %. However, the addition of MPCM will weaken the tensile strength of the potting adhesive. Adding MPCM and h-BN can both effectively improve the thermal conductivity of the potting adhesive: when the filler mass fraction is lower than 20 %, the potting adhesive with MPCM filler exhibits more strengthening capability than h-BN type; while with the continuous increase of filler mass fraction, the thermal conductivity of the potting adhesive with h-BN filler is better. The thermal buffering capacity of the potting adhesive significantly increases with the mass fraction of MPCM, while the effect of h-BN on thermal buffering capacity is not significant. In addition, the addition of h-BN and MPCM significantly improves the temperature uniformity of the potting adhesive.

1. Introduction

Due to the complexity of operating conditions of electric vehicles, the current load on the insulated gate bipolar transistor chip is constantly changing, which will lead to the high temperature and temperature fluctuation of the chip [1]. Excessive temperature and temperature fluctuations can greatly shorten the lifespan of chips [2–4]. Therefore, the thermal management of power components such as Insulated Gate Bipolar Transistor (IGBT) is very necessary. At present, various cooling methods have been applied on high-power electronic devices mainly including air cooling [5], liquid cooling [6] and heat pipe cooling [7], etc.

Due to the range anxiety of electric vehicles, it is very crucial to improve the usage efficiency of electric energy [8]. In recent years, with the further development of phase change materials (PCM), some scholars have applied passive absorption phase change cooling to the cooling of high-power electronic devices [9–11]. Nicholas et al. [12] applied metal as PCM to chip heat dissipation with high pulse power, and proved through experiments that PCM have excellent thermal buffering performance under high pulse power. Ahmad mustaffar et al. [13] proposed to use irregular aluminum foam/brine mixture PCM composite to provide transient cooling and have

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good thermal buffering performance. Hoang et al. [14] found that the thermal buffering performance was far higher by encapsulating the PCM in the shell on the board than by using the traditional standard PCM cardboard. Wang et al. [15] made MPCM by using monodisperse graphene-based aerogel microspheres and phase change wax and verified their excellent stability and high latent heat through experiments. Jagadeeswara et al. [16] prepared the phase change material epoxy resin composite by injecting the PCM into the porous carrier, so that it has a stable shape and improves the thermal buffering capacity. Hao et al. [17] applied PCM to the molybdenum plate of IGBT and found that under different power shocks, the temperature rise rate of the chip was reduced and the temperature fluctuation was reduced. These studies have demonstrated that the application of PCM can effectively improve the heat dissipation capacity of electronic devices.

However, the heat absorbed by PCM is limited. Once the latent heat of the PCM is exceeded, it will hinder heat dissipation [18]. Because the power devices of electric vehicles produce a great deal of heat during operation, it is impossible to satisfy the heat dissipation demand by relying on phase-change cooling alone. Therefore, phase-change cooling often plays an auxiliary role in the thermal management system [19,20]. Some scholars proposed to combine phase change cooling with other cooling methods to improve the heat exchange efficiency of the cooling system [21–24]. Ravindra et al. [25] combined air cooling with phase change cooling and applied it to the heat dissipation of the battery module. The results showed that the maximum temperature rise of the battery module under the 4C discharge rate is less than 5 °C, and the maximum temperature difference is less than 0.12 °C. Similarly, Gao et al. [26] combined liquid cooling and phase change cooling and proved their effectiveness through experiments and simulation. The results showed that the maximum temperature decreased by 33.4 % and the maximum temperature difference decreased by 32.2 %.

Some studies have reported the application of PCMs in reducing contact thermal resistance [27–29], and concluded that PCMs can effectively reduce the contact thermal resistance and improve the heat transfer performance [30–32]. Power devices in electric vehicles are generally encapsulated in potting glue, which has low thermal conductivity. Therefore, most of the heat is transmitted through the substrate [33]. Improving the heat conduction and storage capacity of potting adhesive is also a new way to improve the heat dissipation capacity of power devices.

This study first prepared MPCM and characterized its performance. Subsequently, samples of MPCM/h-BN composite potting adhesive were prepared, and its mechanical properties were tested through mechanical testing. In addition, a thermodynamic testing platform was established to experimentally study the thermodynamic properties of the composite potting adhesive.

2. Experimental systems and data processing

2.1. Materials preparation

The two-component epoxy potting adhesive is YH-A/B adhesive produced by Guangzhou Platinum Bridge Electronic Materials Co., Ltd. The wall material of MPCM is methyl methacrylate (MMA), and the core material is n-docosane, provided by Shaanxi Xihua Chemical Industry Co., Ltd. Sodium dodecyl sulfate (SDS), azodiisobutyronitrile (AIBN), and pentaerythritol tetraacrylate (PETRA) are produced by Nanjing Jiaji New Materials Technology Co., Ltd. Hexagonal boron nitride nano-powder was purchased from Suzhou Napu Material Technology Co., Ltd., with a purity of 99.9 % and an average particle size of 100 nm. KH-560 silane coupling agent was purchased from Nanjing Aocheng Chemical Co., Ltd.

2.2. Synthesis of phase change microcapsule

The suspension polymerization method was chosen in this work to prepare MPCM. Firstly, dissolve 0.96 g of emulsifier SDS in 100 g of deionized water as the aqueous phase. Then, weigh 7.00 g MMA as the wall material, 0.30 g AIBN as the initiator, and 10 g n-dodecane was used as the core material, and 3.00 g of PETRA was used as the crosslinking agent. The oil phase was prepared by mixing and dispersing with ultrasound for 20 min. Mix the prepared water phase and oil phase into a 250 mL flask and stir at 35 °C at 3000 r/min for 20 min to form a stable oil in water lotion. Put the dispersed lotion in a water bath at 80 °C, and stir it for 2 h at a speed of 600 r/min. Finally, the obtained lotion was filtered by suction, washed three times with ethanol, and dried for 24 h in a 40 °C electric blast constant temperature drying oven to prepare MPCM.

2.3. Characterization of phase change microcapsules

A particle size analyzer (Mastersizer 3000) is used to analyze the particle size distribution of the prepared MPCM. The phase change temperature and latent heat of MPCM were measured by using a differential scanning calorimeter (DSC 8500) under N₂ atmosphere. The temperature range was 15~200 °C, the temperature rise rate was 10 °C/min, and the measurement accuracy was ±0.01 °C. The thermal stability and composition of MPCM were characterized using a thermogravimetric analyzer (Pyris 1 TG A) under N₂ atmosphere. The temperature range tested was 15–800 °C, the temperature rise and fall rate was 10 °C/min, and the measurement accuracy was ±0.01 mg. The encapsulation efficiency of phase change microcapsules can be calculated using the enthalpy of phase change measured by differential scanning calorimetry (DSC). The encapsulation rate E_e (%) is the ratio of the content of PCM in MPCM to the designed amount of PCM, as shown:

$$E_e = \frac{c_a}{c_t} \times 100\% \quad (1)$$

where, c_a is the actual core mass, and c_t is the total mass of MPCM.

2.4. Fabrication of potting adhesive samples

Epoxy resin adhesive, MPCMs, and h-BN were added according to the different mass fraction formulas and thoroughly mixed for 30 min in a high-speed shear dispersion machine. The curing agent methylhexahydrophthalic anhydride and accelerator DBU were added, and then thoroughly stirred and ultrasonically dispersed for 30 min. Then pour the mixture into the self-made mold and place it in a vacuum drying oven at 60 °C for an hour for exhaust. After the exhaust is completed, continue to heat up to 100 °C and cure for 2 h. After solidification, slowly cool for demolding, and then cut and ground before use.

2.5. Thermal behavior and mechanical experiments

The universal mechanical testing machine is used to measure the mechanical properties of composite potting adhesive. The tested tensile specimens are all dumbbell shaped with a length of 20 mm, a width of 2.5 mm, and a thickness of 2.0 mm. The mold is shown in Fig. 1.

The thermodynamic performance test module of composite potting adhesive is shown in Fig. 2 (a). The testing principal schema of the thermodynamic performance testing module for composite potting adhesive is shown in Fig. 2 (b). The ceramic heating plate is connected to an adjustable DC stabilized power supply. The thermocouple sensor is arranged in the middle of the ceramic heating plate and connected to the temperature acquisition module and computer. The calibrated infrared imager is used to monitor the temperature of the experimental module outside the incubator. The experimental process is as follows: adjust the temperature of the incubator to 40 °C, place the test module in the incubator and let it sit for 1 h. Considering that the PCM failure temperature is 150 °C, heating is carried out at a constant power of 5W for 100 s. During this period, the entire heating and cooling process on the surface of the heating plate is collected and recorded using devices such as thermocouple sensors. The overall temperature testing platform is shown in Fig. 2 (c).

The infrared imaging device and supporting software are used here to measure the temperature distribution of the composite sealant testing module. In the monitoring interface, relevant temperature monitoring points can be set and real-time temperature distribution maps can be exported.

2.6. Data analysis

In response to the shortcomings of existing testing methods, this experiment designed a testing method for PCM/potting adhesive, defined as Thermal Buffering Capacity (TBC), which reflects the ability of composite potting adhesive to adjust temperature under sudden changes in heat flux.

$$TBC = |T_{\text{control}} - T| \quad (1)$$

where T_{control} is the surface temperature of the heating element in the comparison potting adhesive, and T is the surface temperature of the composite potting adhesive heating element.

From Eq. (2), the thermal buffering capacity TBC_{control} of the composite sealant within a certain temperature range is obtained.

$$TBC_{\text{control}} = \int_{T_2}^{T_1} (T_{\text{control}} - T) dT \quad (2)$$

where, T_1 is the starting position of the temperature range, T_2 is the endpoint position of the temperature range.

Due to the inherent errors of the sensors used in the experiment, the measurement data will be affected by these inaccuracies. The error of thermocouple is ± 1 °C, the error of infrared imager is ± 1 °C, and the error of tensile strength is 5 %.

3. Results and discussions

Fig. 3(a) shows the distribution of MPCM particle size. From the figure, it can be found that the particle size distribution of the MPCM is relatively uniform, and the average particle size is about 10 μm . Fig. 3(b) shows the DSC curve of MPCM. From the DSC experimental results, in the initial stage, DSC tends to be horizontal, which is because there is no phase transition at this moment. Therefore, the specific heat changes very little with temperature. The peak phase transition temperature of PCM is 45.4 °C, the Onset in

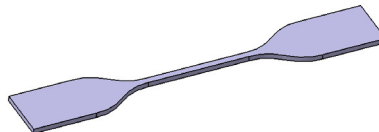


Fig. 1. Sample for tensile testing.

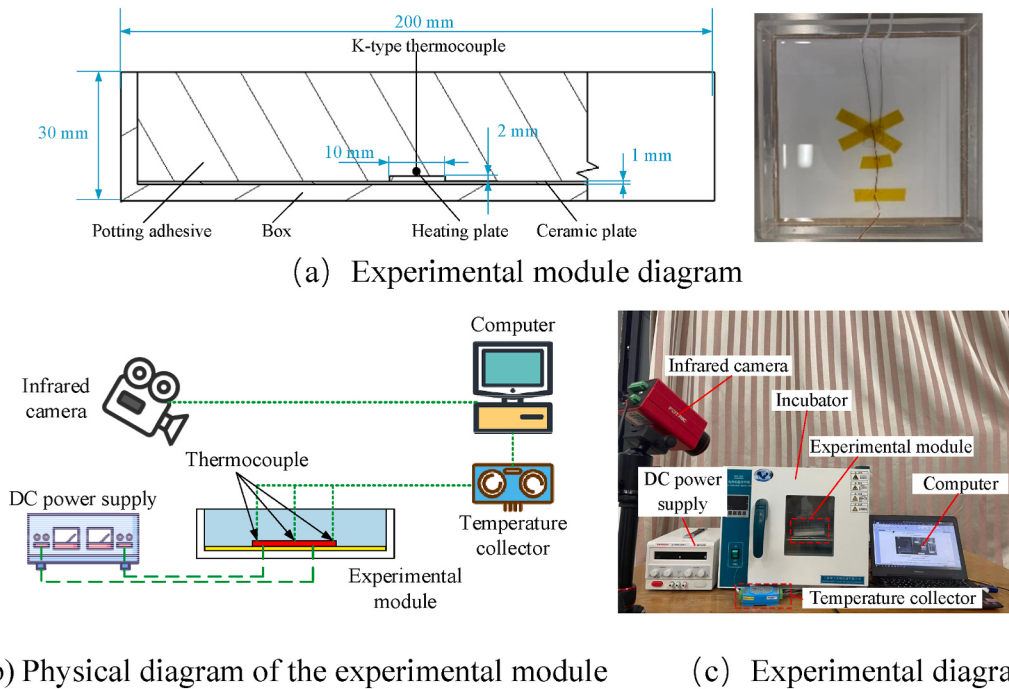


Fig. 2. Field schema of temperature test platform.

the figure represents the starting temperature of phase transition at 41.0°C , and End set represents the ending temperature of phase transition at 46.7°C . They are the intersection points of the phase transition peak temperature and the solid-liquid phase baseline as a straight line drawn by the slope of both sides of the DSC curve. Area is the closed area formed by the line connecting the solid-liquid phase baseline and the DSC curve, which is 828.2 mJ . It can be concluded that the phase change latent heat of MPCM is 166.3 J/g , indicating that the MPCM has a good thermal storage performance. Fig. 3(c) and (d) show the thermogravimetric analysis (TGA) and derivative thermogravimetry (DTG) curves of MPCM, respectively. Through the analysis, the heat resistance performance of MPCM can be determined. The weight loss rate of MPCM in the first stage ($15\text{--}150^{\circ}\text{C}$) is 4.1% , and the weight loss curve is flat, mainly due to the escape of adsorbed water. In the second stage ($150\text{--}270^{\circ}\text{C}$), the weight loss rate sharply increased mainly due to the melting of the PMMA shell, which caused the escape of n-docosane inside. The weight loss rate in this stage was 70.1% . The third stage ($270\text{--}425^{\circ}\text{C}$) is mainly caused by the escape and sublimation of the capsule wall material, with a weight loss rate of 21.3% during this stage. In the fourth stage ($425\text{--}800^{\circ}\text{C}$), there is a residual amount, accounting for approximately 4.5% , which can be considered as ash content.

Fig. 4 shows the comparison of tensile strength of potting adhesive with different mass fractions of fillers. The tensile strength can help understand the strain capacity of the potting adhesive under tensile load, so it is crucial for the packaging of IGBT that generates stress. From Fig. 4, it can be found that the addition of MPCM has a slight negative effect on the tensile strength of the potting adhesive. The tensile strength of the content of 40% MPCM composite in potting adhesive decreased by 3.61% . The filling of MPCM is prone to creating gaps within the potting adhesive, resulting in a decrease in its tensile strength. On the contrary, adding an appropriate amount of h-BN can improve the tensile strength. When the mass fraction of h-BN added is 10% , 20% , and 30% , the tensile strength of the potting adhesive is increased by 1.05% , 4.96% , and 7.07% compared to pure potting adhesive, respectively. However, when the mass fraction of h-BN nitride increases to 40% , the tensile strength tends to decrease to 70.1 MPa . The higher the proportion of hexagonal boron nitride, the lower the overall viscosity of the potting adhesive, resulting in a decrease in crosslinking degree. This is the key factor in reducing the tensile strength of composite potting adhesive.

As is well known, the thermal conductivity of ordinary epoxy resin cured products is generally $0.2\text{--}0.3\text{ W/(m}\cdot\text{K)}$. The addition of thermal conductive fillers is worth of being investigated to enhance the thermodynamic performance of the potting adhesive. MPCM and h-BN were selected as different thermal conductive fillers for potting adhesive and its thermal conductivity for h-BN with different mass fractions are illustrated in Fig. 5. From the figure, it can be seen that as the mass fraction of h-BN is low, the thermal conductivity of the potting adhesive improves little. When the mass fraction of h-BN reaches 10% , the thermal conductivity of the potting adhesive only increases by $0.005\text{ W/(m}\cdot\text{K)}$. This is mainly due to the amount of thermal conductive fillers added is too small and interaction and crosslinking between the fillers cannot be formed. Therefore, at this point, the thermal conductivity is still determined by the characteristics of the potting adhesive itself. When the filling amount of h-BN reaches 20% , the fillers interact with each other to form a thermal conductivity network, achieving the goal of improving its thermal conductivity. But when the filling mass fraction reaches 30% , the increase in thermal conductivity value is limited, the content increases by 10% , and the thermal conductivity only increases by $0.017\text{ W/(m}\cdot\text{K)}$. From the figure, it can be seen that within a limited range, the thermal conductivity of the encapsulation adhesive increases with the increase of MPCM. It improves the thermal conductivity of the potting adhesive by

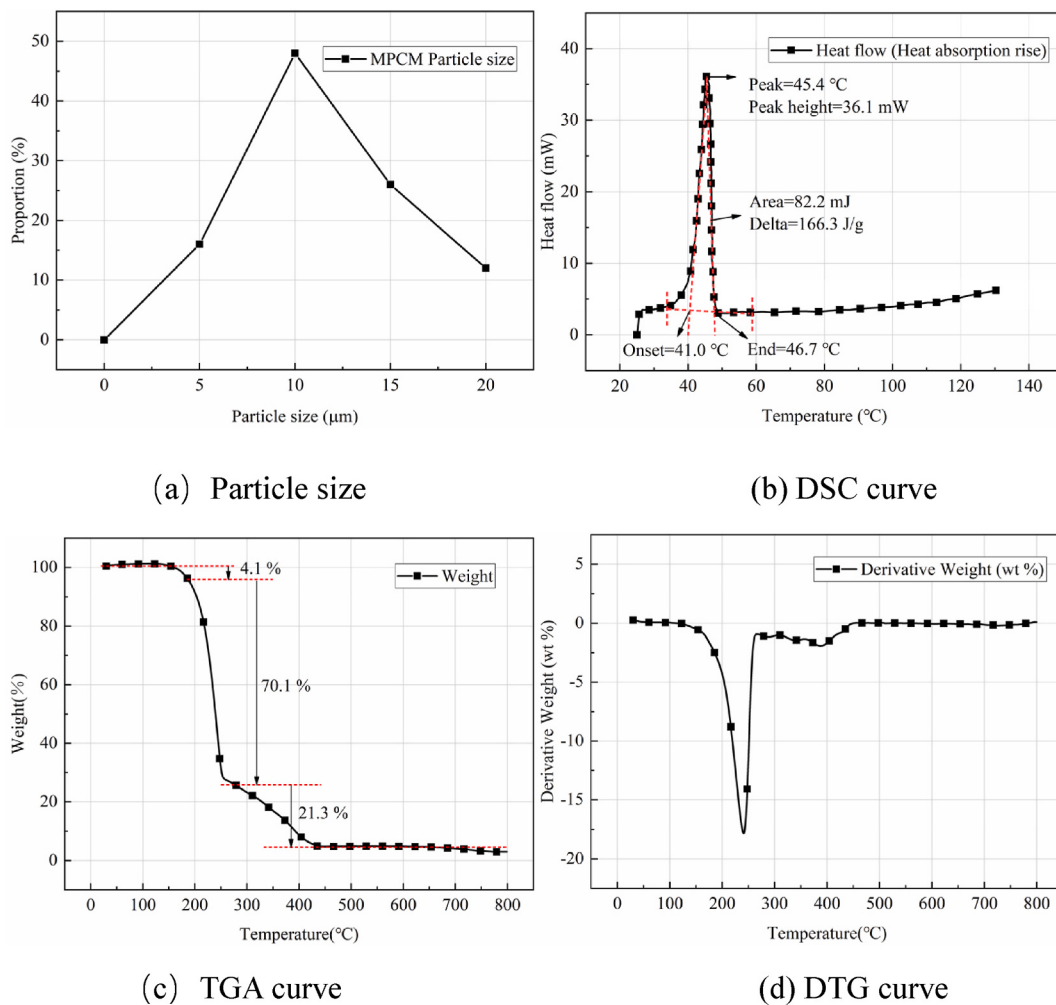


Fig. 3. Characterization of MPCM.

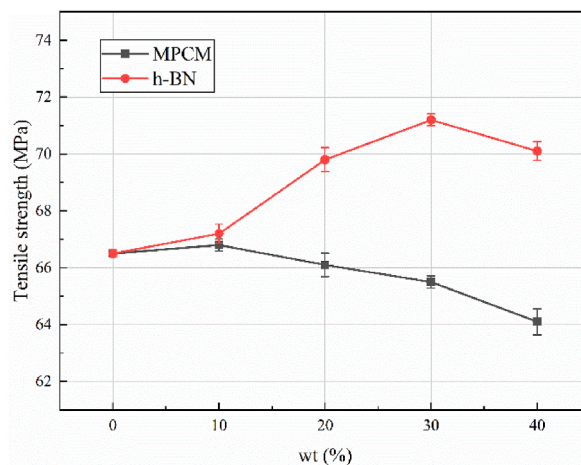


Fig. 4. Comparison of tensile strength of composite potting adhesive.

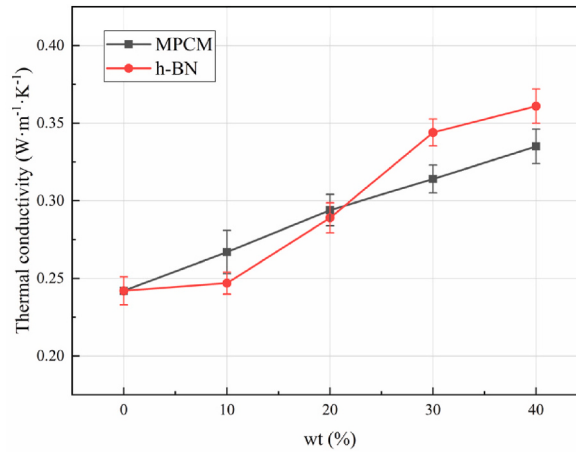


Fig. 5. Comparison of thermal conductivity of composite potting adhesive.

absorbing heat through MPCM, so overall it shows a linear increase. When the mass fraction of MPCM is 40 %, the thermal conductivity of the potting adhesive reaches 0.0335 W/(m·K). When the MPCM is below 20 %, the heat transfer performance of the MPCM is better, but with the increase of filler content, the heat transfer performance of h-BN is better.

Fig. 6 shows the temperature changes on the surface of a heating element with a heating power of 5 W for different mass fractions of MPCM encapsulation adhesive. Considering the working temperature limit of phase change microcapsules, heating should be stopped when the surface temperature of the heating element reaches to 150 °C. It can be found that the heating plate temperature in the MPCM encapsulation adhesive is slower in the initial heating rate compared to the pure one, and the higher the mass fractions, the longer the slowing down area. When the temperature rises to 60 °C, the temperature rise rate of the heating plates in each test block is basically similar. This is due to during the initial heating stage, the MPCM in the potting adhesive can absorb some of the heat through the MPCM, causing a slower rate of temperature rise. When the phase transition temperature is exceeded, it is no longer possible to absorb heat through the phase transition, and the rates of temperature rise are similar. Similarly, the higher the mass fractions of MPCM, the greater the amount of heat that can be absorbed through phase change.

Fig. 7 shows the temperature changes on the surface of a heating element with 5W heating power for different contents of hexagonal boron nitride potting adhesive. From the figure, it can be seen that 10 % hexagonal boron nitride has little effect on the heat dissipation ability of the potting adhesive. This is because the amount of h-BN is too small to form a thermal conductivity grid in the potting adhesive. Subsequently, at a hexagonal boron nitride content of 10 %, 20 %, 30 %, and 40 %, the time required to heat the surface of the heating element to 150 °C was extended by 4.12 %, 12.1 %, 24.1 %, and 49.6 %, respectively. This is because the thermal conductivity of h-BN is much higher than that of the potting adhesive itself, which can effectively improve the overall thermal conductivity of the potting adhesive and enable faster heat dissipation.

Fig. 8 shows the comparison of thermal buffering performance of potting adhesive under different ratios. From the figure, it can be known that the thermal buffering ability of the potting adhesive significantly increases with the increase of MPCM mass fractions. The thermal buffering capacity of the composite potting adhesive with 10 %, 20 %, 30 %, and 40 % MPCM content is 1310.726, 3144.373,

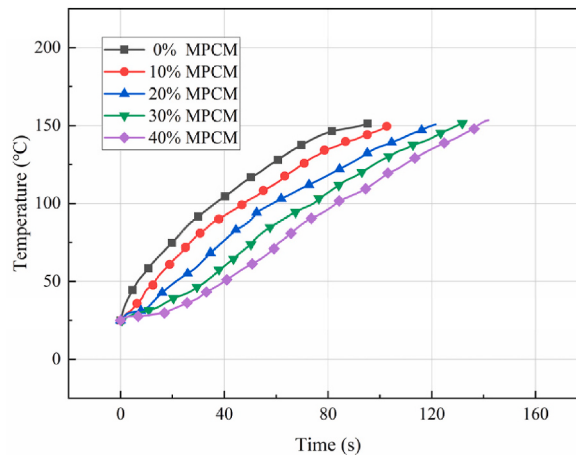


Fig. 6. Temperature variation of composite potting adhesive with MPCM in different mass fractions.

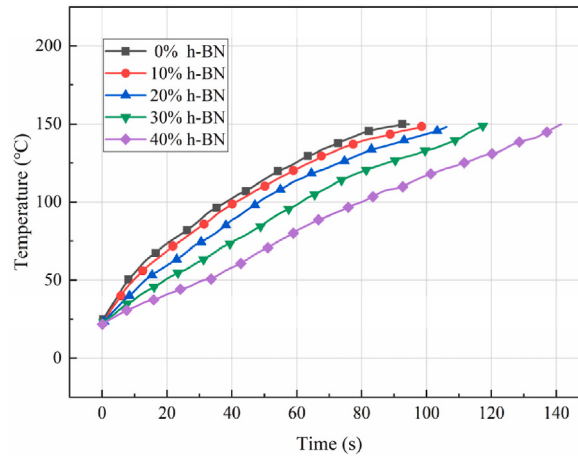


Fig. 7. Temperature variation of composite potting adhesive with h-BN in different mass fractions.

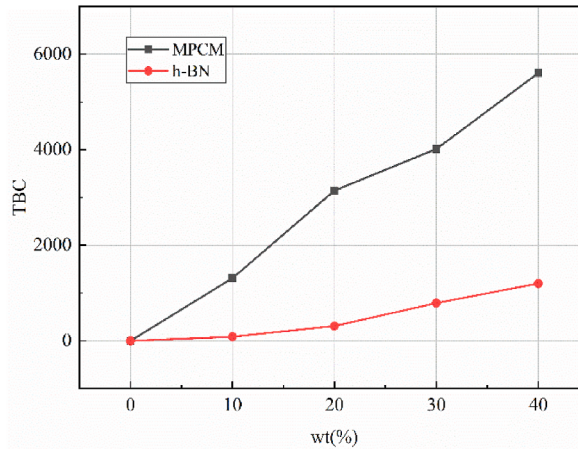


Fig. 8. Comparison of thermal buffering capacity of composite potting adhesive with MPCM or h-BN.

4018.762, and 5608.872, respectively. However, the effect of h-BN on thermal buffering ability is not significant. At the same 40 % mass ratio, its thermal buffering ability is only 21.4 % of that of MPCM composite potting adhesive. MPCM absorbs heat that cannot be discharged in a timely manner through a high latent heat value. H-BN can only accelerate the speed of heat transfer and delay the time when the temperature peak reaches.

Fig. 9 shows the comparison of the maximum temperature difference of potting adhesive at different mass fractions when reaching 150 °C at a heating power of 5 W. From the graph, it can be found that as the mass fraction of fillers increases, the temperature uniformity of the potting adhesive improves. This can be explained by the thermal diffusion rates. Among them, the improvement effect of h-BN at 10 % is not significant, because the content of h-BN at this moment is not enough to form a thermal conductivity network, so there is not much improvement in the thermal conductivity coefficient. After the mass fraction reaches 20 %, the thermal conductivity significantly increases, resulting in a significant reduction in temperature difference. In the composite potting adhesive of MPCM, the temperature difference is reduced by absorbing the heat transferred by the heating element through MPCM in a linear way, but as the PCM completely melts, the improvement of thermal diffusion performance by MPCM will reach its limit.

4. Conclusions

The influences of MPCM and h-BN on the mechanical and thermodynamic properties of composite potting adhesive were studied through experimental data. MPCM and h-BN composite potting adhesive with mass fractions from 10 % to 40 % were prepared separately, and their tensile strengths were tested by using a universal mechanical testing machine. In addition, the thermodynamic properties of composite potting adhesive with different filler ratios were studied. The main conclusions are.

- (1) h-BN has a positive effect on the tensile strength of composite potting adhesive, with the best tensile strength at a mass fraction of 30 %, which is 71.2 MPa, with an increase of 7.1 % compared to the original potting adhesive. However, MPCM has a

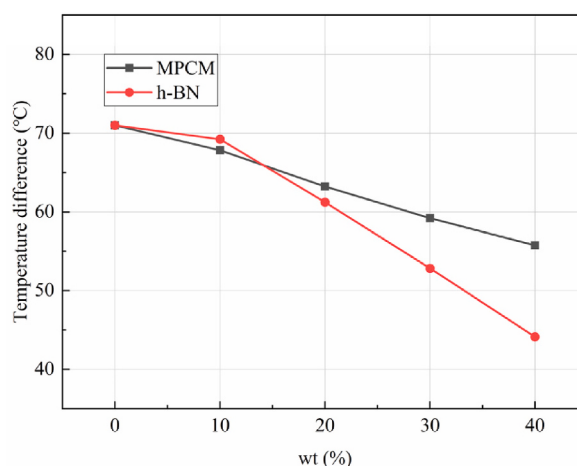


Fig. 9. Comparison of maximum temperature difference.

negative effect on the tensile strength of the potting adhesive, with tensile strengths of 66.5, 66.8, 66.1, 65.5, and 64.1 MPa at 10 %, 20 %, 30 %, and 40 %.

- (2) The thermal buffering capacity of the composite potting adhesive significantly increases with the increase mass fraction of MPCM in a linear way. However, the effect of h-BN nano powder on thermal buffering capacity is not significant. At the 40 % mass fraction, its thermal buffering capacity is only 21.4 % of that of MPCM potting adhesive.
- (3) h-BN and MPCM can be used as the thermal conductive fillers to effectively improve the thermal conductivity of the potting adhesive. When the filler content is less than 20 %, the heat transfer performance of the MPCM is better, but as the filler content continues to increase, the heat transfer performance of the h-BN composite potting adhesive is better. In addition, with the increase of the mass fraction of h-BN and MPCM, the temperature uniformities of the encapsulation adhesive are both improved.

CRedit authorship contribution statement

Jing Xu: Resources, Formal analysis, Yunquan Zhu, Formal analysis. **Xiang Wang:** Writing – review & editing, Writing – original draft, Supervision. **Meng Zhang:** Data curation.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgement

This work was supported by National Natural Science Foundation of China (GrantNo.52306167), Six talent peaks project of Jiangsu Province (GrantNo.2015-ZBZZ-035).

Data availability

Data will be made available on request.

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